

**ANNUAL REPORT
TO
NATIONAL AERONAUTICS
AND
SPACE ADMINISTRATION FOR
CHARACTERIZATION OF
PHYSIOCHEMICAL PROPERTIES
OF
POLYMERIC AND ELECTROCHEMICAL
MATERIALS FOR AEROSPACE FLIGHT**



National Aeronautics and
Space Administration

GODDARD SPACE FLIGHT CENTER

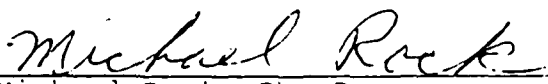
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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
FOR
CHARACTERIZATION OF PHYSIOCHEMICAL PROPERTIES OF
POLYMERIC AND ELECTROCHEMICAL MATERIALS FOR AEROSPACE FLIGHT

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ABSTRACT

Nickel-cadmium rechargeable batteries are a vital and reliable energy storage source for aerospace applications. As the demand for longer life and more reliable space batteries increases, the understanding and solving of cell aging factors and mechanisms become essential.

Over the years, many cell designs and manufacturing process changes have been developed and implemented. Cells fabricated with various design features were life cycled in a simulated low-Earth orbit regime. Following the test program, a comprehensive electrochemical analysis of cell components was undertaken to study cell degradation mechanisms.

Discharge voltage degradation or voltage plateau has been observed during orbit cycling, but, its cause and explanation have been the subject of much discussion. A Hg/HgO reference electrode was used to monitor the reference versus each electrode potential during the discharge of a cycled cell. The results indicate that the negative electrode was responsible for the voltage plateau. Cell analysis revealed large crystals of cadmium hydroxide on the surface of the negative electrode and throughout the separator.

The migration of active material from the negative electrode into the separator during cycling has been identified as a contributing factor causing the loss of negative plate efficiency and capacity. Preliminary laboratory results indicate that the rate of cadmium migration is affected by temperature, charge rate, and charge duration.

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DESIGN VARIABLE CELL ANALYSES

During the past two decades, various cell design and manufacturing process changes have been implemented for the purpose of improving the performance of the nickel-cadmium cell for space application. Various users have tested and evaluated these cells, but a systematic approach for comparing the relative merits of each design or process change does not exist.

The National Aeronautics and Space Administration's (NASA) Goddard Space Flight Center (GSFC) in conjunction with General Electric embarked on a program to evaluate eight of the more important cell design variations. The details of the cell designs are described in a paper presented at the Electrochemical Society Conference in 1978 by Floyd Ford and Dave Baer. The test cells were fabricated and assembled into cell packs and life cycled in a low-Earth orbit cycle regime. Orbit parameters are given in the previously mentioned reference.

The test program was terminated after 4 years and a tear-down analysis of 12 selected cells was undertaken. The following outline summarizes the physical and electrochemical analyses performed on these cells:

a. Electrolyte

- Hydroxyl and carbonate content

b. Separator

- Physical
- Electrolyte and cadmium hydroxide content

c. Negative Electrode

- Physical
- Charge and discharge active material content
- Additive content

d. Positive Electrodes

- Physical
- Charge and discharge active material content
- Additive content

At present, only four cells have completed the analysis program. Tables 1 through 4 show the typical results of the cell analysis. Results of all the analyses will be documented in a NASA Technical Memorandum.

VOLTAGE DEGRADATION AND PLATEAU

INTRODUCTION

Nickel-cadmium cells are used extensively in low-Earth orbit satellite power systems, with age, and have been observed to show a voltage degradation during discharge. An understanding of the electrochemical mechanism responsible for the discharge voltage plateau is useful in the development of batteries with higher energy density and greater reliability.

Table 1
Control Cell Group
(Cell 006)
Positive Electrode

Plate Composition (by weight (wt))	Plate 2	Plate 8	Plate 12
NiOOH--charged (g)	0.9648	1.3581	1.0029
Ni(OH) ₂ --discharged (g)	5.0504	4.1881	5.3014
Co(OH) ₂ (g)	0.3073	0.3112	0.3199
Cd(OH) ₂ (g)	0.9065	0.8864	0.9293
Ni in plaque (g)	3.3876	3.1479	3.2528
Substrate (g)	2.6724	2.6888	2.6298
Nickel tab (g)	0.06	0.06	0.06
Total wt (g)	13.349	12.6405	13.4961
Actual wt (g)	13.808	13.578	13.822
% Deviation	3.324	6.905	2.358
Dimensions			
Length in cm	7.62	7.62	7.62
Width in cm	6.9454	6.9454	6.9454
Thickness in cm	0.07856	0.07856	0.07856
Solid volume, cc	4.16	4.16	4.16
Density of nickel (g/cc)	0.8143	0.7567	0.7819
% Porosity (incl substrate)	83.63	84.24	84.11
Effective substrate thickness (in)	0.0024	0.0024	0.0024
% Effective Porosity (excl substrate)	90.85	9.150	9.124
Volume of voids, cc	3.7773	3.8061	3.796
Nickel active material/cc void	0.0159	0.01457	0.01661
Total active material/cc void	2.946	2.8610	2.945
% Co(OH) ₂ in active material	2.76	2.858	2.858
% Cd(OH) ₂ in active material	8.14	8.14	8.30
Total active material (g)	11.1356	10.8892	11.1922

Table 2
Control Cell Group
(Cell 006)
Negative Electrode

Plate Composition (by weight (wt))	Plate 3	Plate 9
Cd(OH) ₂ (g)	8.8260	8.5129
Cadmium metal (g)	0.1809	0.1915
Ni(OH) ₂ (g)	0.6285	0.6112
Ni in plaque (g)	4.3898	4.5065
Substrate (g)	2.9418	2.9586
Nickel tab (g)	0.0600	0.0600
Total wt (g)	17.0270	16.8407
Actual wt (g)	16.9810	16.977
% Deviation	0.27	0.80
Dimensions		
Length in cm	7.62	7.62
Width in cm	6.9454	6.9454
Thickness in cm	0.0829	0.0829
Solid volume, cc	4.3874	4.3874
Density of nickel (g/cc)	1.0064	1.027
% Porosity (incl substrate)	81.22	80.88
Effective substrate thickness (in)	0.0024	0.0024
% Effective Porosity (excl substrate)	88.69	88.46
Volume of voids, cc	3.8913	3.8811
Cadmium active material/cc void	2.3286	2.2577
Total active material/cc void	2.5228	2.4002
% Ni(OH) ₂ in active material	6.523	6.561
Total wt of active material	9.6354	9.3156

Table 3
Control Cell Group
Without Positive PQ Treatment
(Cell 003)
Positive Electrode

Plate Composition (by weight (wt))	Plate 2	Plate 8	Plate 12
NiOOH--charged (g)	0.6628	0.8476	1.2204
Ni(OH) ₂ --discharged (g)	7.6418	7.2752	7.4892
Co(OH) ₂ (g)	0.1938	0.2093	0.1947
Cd(OH) ₂ (g)	0.8431	0.8372	0.8344
Ni in plaque (g)	1.3213	1.3557	1.3314
Substrate (g)	2.8298	2.6868	2.6735
Nickel tab (g)	0.06	0.06	0.06
Total wt (g)	13.5526	13.1718	13.7436
Actual wt (g)	13.9080	13.8690	13.7990
% Deviation	2.62	5.02	0.4
Dimensions			
Length in cm	7.62	7.62	7.62
Width in cm	6.9454	6.9454	6.9454
Thickness in cm	0.08293	0.08293	0.08293
Solid volume, cc	4.39	4.39	4.39
Density of nickel (g/cc)	0.3009	0.3088	0.3032
% Porosity (incl substrate)	89.375	89.653	89.750
Effective substrate thickness (in)	0.0024	0.0024	0.0024
% Effective Porosity (excl substrate)	96.619	96.530	96.593
Volume of voids, cc	4.2416	4.2377	4.2404
Nickel active material/cc void	0.1955	0.1966	0.0254
Total active material/cc void	2.612	2.639	2.624
% Co(OH) ₂ in active material	1.749	1.7984	1.7500
% Cd(OH) ₂ in active material	6.2501	7.421	7.4998

Table 4
Control Cell Group
Without Positive PQ Treatment
(Cell 003)
Negative Electrode

Plate Composition (by weight (wt))	Plate 3	Plate 9
Cd(OH) ₂ (g)	6.3112	6.5445
Cadmium metal (g)	1.7514	1.8382
Ni(OH) ₂ (g)	0.5413	0.6026
Ni in plaque (g)	3.8954	4.2239
Substrate (g)	2.6894	2.5437
Nickel tab (g)	0.06	0.06
Total wt (g)	15.2478	15.8130
Actual wt (g)	14.8505	15.1064
% Deviation	2.227	4.28
Dimensions		
Length in cm	7.6200	7.6200
Width in cm	6.9454	6.9450
Thickness in cm	0.10859	0.10859
Solid volume, cc	5.75	5.75
Density of nickel (g/cc)	0.6774	0.7346
% Porosity (incl substrate)	87.13	86.77
Effective substrate thickness (in)	0.0024	0.0024
% Effective Porosity (excl substrate)	92.39	91.74
Volume of voids, cc	5.3124	5.2750
Cadmium active material/cc void	0.01518	0.01589
Total active material/cc void	2.289	2.382
% Ni(OH) ₂ in active material	4.4511	4.7967

Different views have been expressed as to which electrode and which mechanism is responsible for the voltage loss. One mechanism (Reference 1) theorizes that the recrystallization of cadmium hydroxide at the surface of the cathode during cycling insulates the lower layers of active cadmium and causes the discharge to occur at a very low rate. Barnard et al. (Reference 2) report that an alloy, $\text{Ni}_5\text{Cd}_{21}$, formed in the negative electrode of cells and subjected to continuous charging at elevated temperatures causes voltage degradation. Russian workers (Reference 3) report that under certain conditions (e.g., if the battery is stored in the charged state or cycled with incomplete discharge) some of the cadmium forms the intermetallic compound $\text{Ni}_5\text{Cd}_{21}$ with nickel, and is discharged at a potential on the positive side of the usual potential by 0.1 V. Recently, Barnard et al. (Reference 4) reported that the secondary discharge plateau is associated with the inefficient reduction of sintered plate NiOOH electrodes. They point out that the potential of the lower plateau is highly dependent on discharge rate and to some extent on the charge regime applied to the electrode. Zimmerman and Janecki (Reference 5) also report that voltage losses occur as a result of changes in the nickel electrode potential with cycling.

Gerald Halpert (Reference 6) presented the results of periodic capacity discharges performed on 26.5-Ah cells during the life cycle test program. These cells, which were cycled in a low-Earth orbit regime at 10°C and to a voltage limit, showed voltage degradation after several thousand cycles. Figure 1 shows the voltage plateau behavior of cells from pack 26 H which were cycled at 25 percent depth of discharge (DOD).

EXPERIMENTAL METHODS

A Hg/HgO electrode served as a stable reference electrode and was used to monitor the reference potential versus the negative and positive electrode of the cell being tested. This electrode

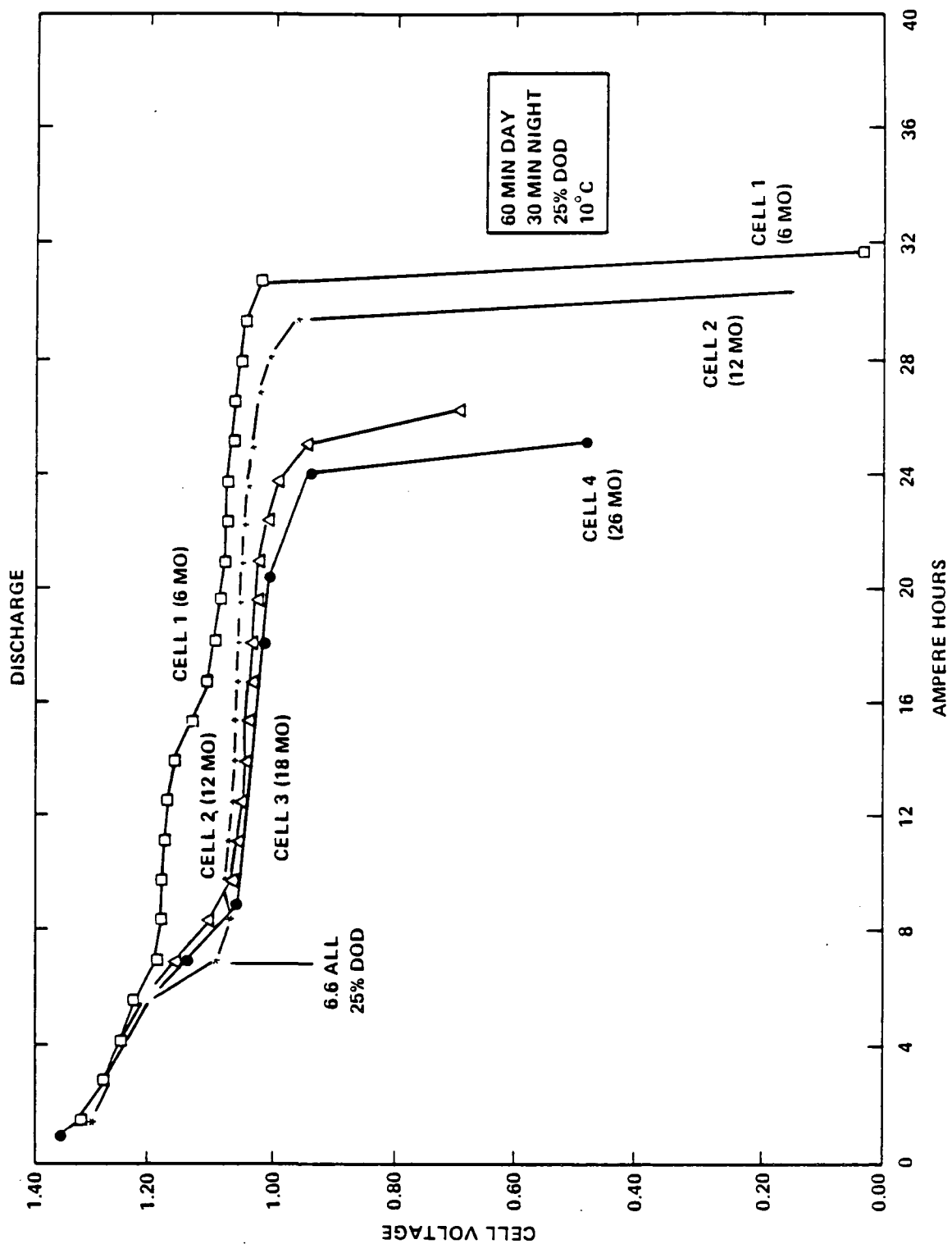


Figure 1. Capacity Test for Pack 26 H

consisted of small amounts of mercury and mercuric oxide mixed with two to three drops of 31 percent potassium hydroxide (KOH) and placed in a Teflon tube of 50-mm diameter with a tiny hole at the bottom that was filled with separator material. Several millimeters of pure mercury were placed over the Hg/HgO. A platinum wire was immersed in the mercury pool as precautions were taken to prevent the wire from coming into contact with the Hg/HgO interface.

Table 5 shows the history of the cells that were used in this experiment. Initially, a 20-Ah cell from pack 12 F was chosen for investigation because it had undergone 24,958 cycles in a low-Earth orbit regime at 10°C and 40 percent DOD.

The fully charged cell was discharged to 50 percent and the Hg/HgO reference electrode was carefully introduced by drilling a hole on the top of the cell and sealing the hole all around with putty. Care was taken to prevent the cell from exposure to air by keeping it in a polyethylene bag filled with nitrogen. The cell was recharged to the voltage limit of 1.458 V and discharged at 10 A rate to 0.5 V.

During the discharge, a two-channel recorder monitored and recorded the reference versus negative and reference versus positive electrode potentials. Cell voltage was recorded separately. Figure 2 shows the results that were obtained from the discharge of the cell from pack 12 F. The discharged cell, which was again cycled 10 times and was discharged at 10 A, continued to monitor the potentials of the negative and the positive versus the Hg/HgO reference. The cell did not exhibit the second voltage plateau.

Similar experiments were conducted using three 12-Ah cells one each from pack 3H, 3J, and 3D and another 20-Ah cell from pack 1 K. These cells were discharged at Crane, Indiana using the reference electrode technique described earlier. Typical results shown in Figure 2 show that the negative electrode was responsible for the second voltage plateau.

Table 5
Cell History and Operating Parameters

Pack number	12F	1K	3H	3J	3D
Cell number	3	1	3	3	2
Nominal cap (Ah)	20	20	12	12	12
Vol. of KOH (ml)	85	85	41.5	39	40
Pos. loading (g/dm ³)	1704	1704	2113	2130	2095
Neg. loading (g/dm ³)	1865	1865	2180	2542	2180
Special treatment	NEG	PELLON	No PQ	Old proc.	Control
Orbit period (HR)	TEFLNTD	2503	Treat	no PQ	group
Temp (°C)	1.5	1.5	1.5	1.5	1.5
Charge rate (A)	10	20	20	20	20
No. of cycles	16	16	9.6	9.6	9.6
DOD (%)	24,958	18,864	23,281	23,334	23,467
Volt. limit (V)	40	40	40	40	40
Ah-out to 0.5 V (Ah)	1.457	1.434	1.453	1.453	1.453 to 1.473
Pos. thickness (in)	14.50	10.86	8.19	8.80	8.35
Neg. thickness (in)	0.027	0.027	0.027	0.032	0.027
	0.0315	0.0315	0.031	0.026	0.031

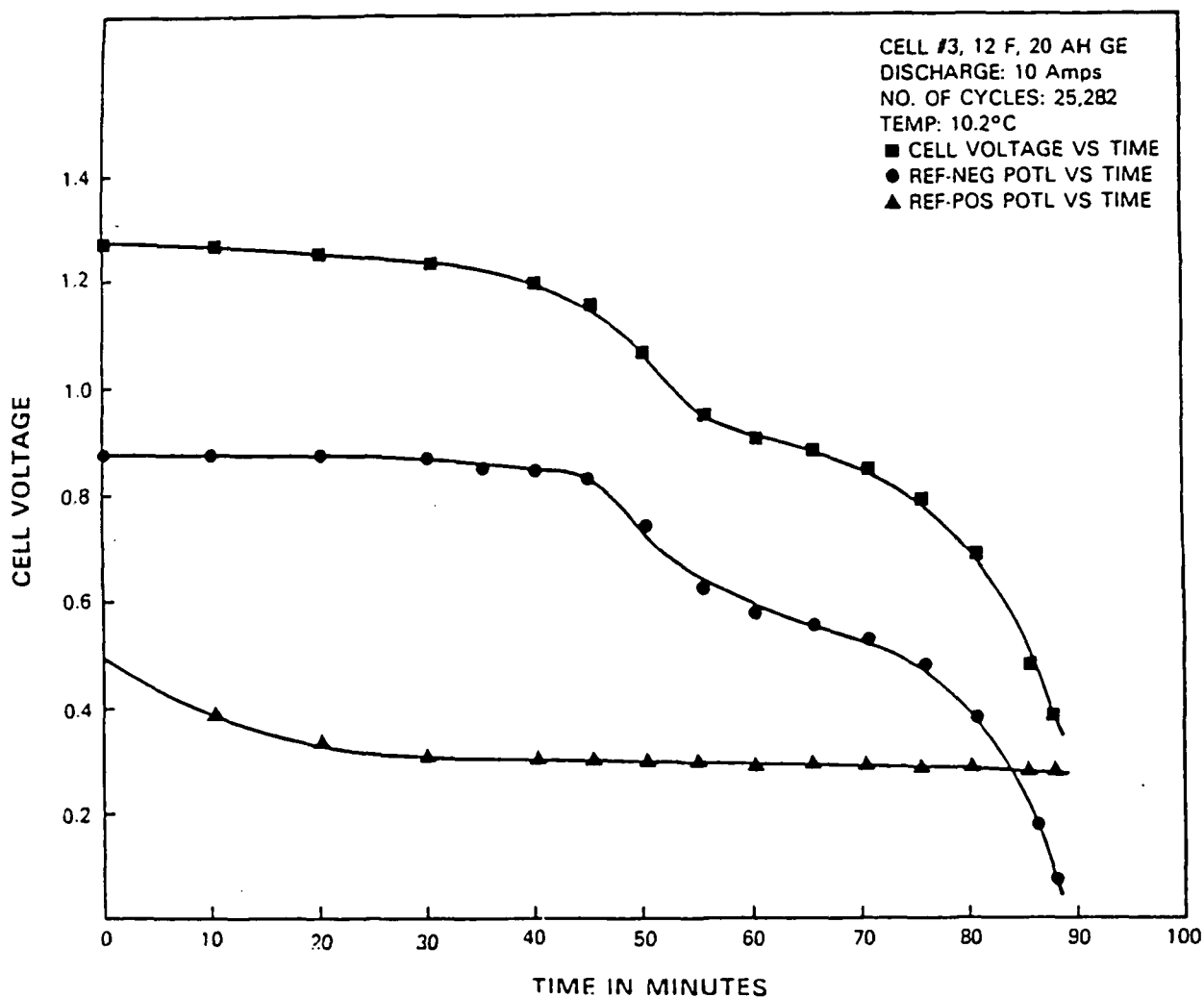


Figure 2. Cell Discharge and Reference Electrode Voltage Profiles

RESULTS AND DISCUSSION

Results of the present investigation indicate that the negative electrode is responsible for the second voltage plateau in nickel-cadmium cells. Figure 3 is the reference voltage versus each electrode for one cell in pack 1 K. Comparing the positive versus reference electrode of Figure 3 to the negative versus reference electrode (Hg/HgO) profile in Figure 2, it is evident that the magnitude of the voltage plateau with respect to the negative

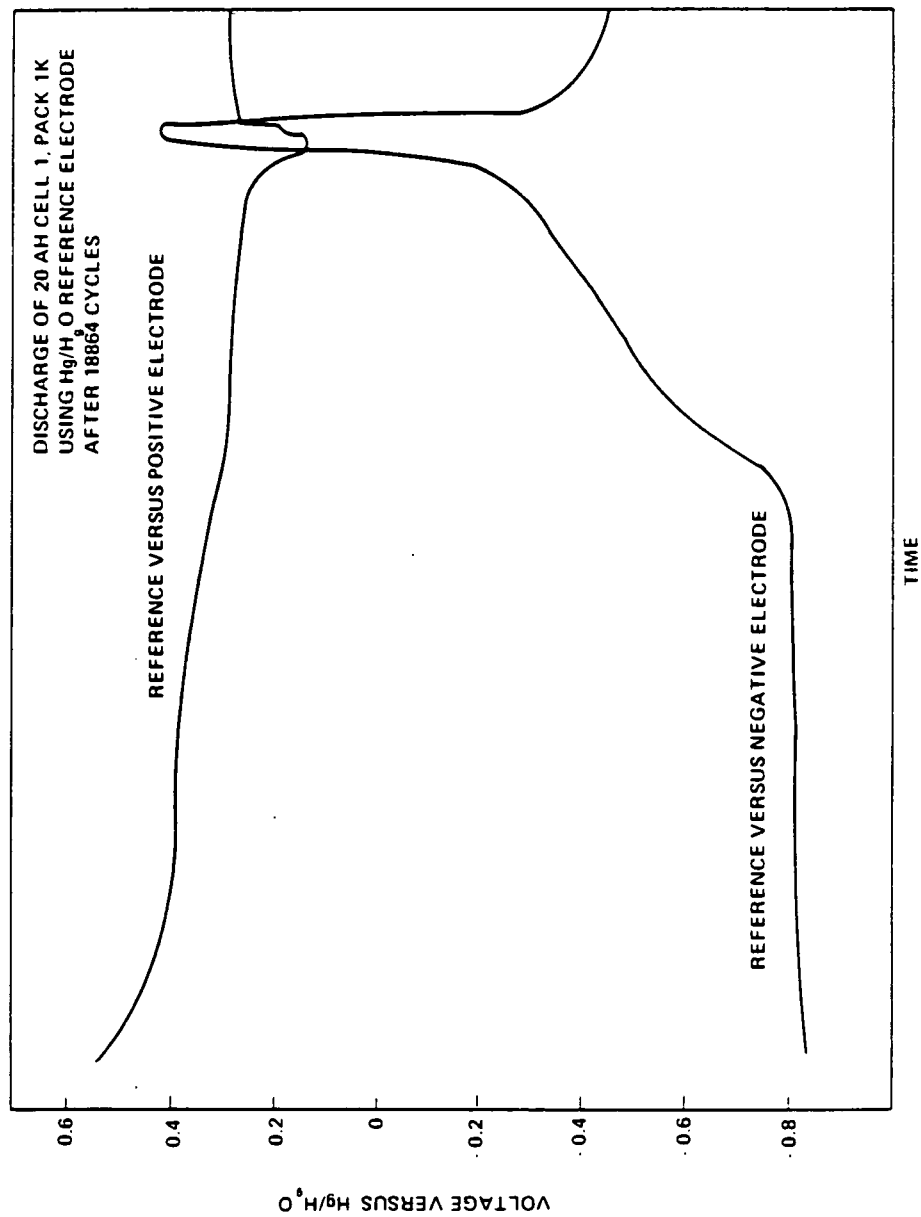
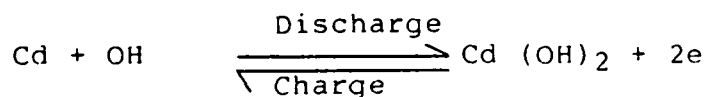


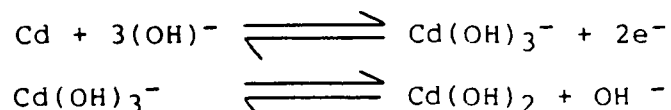
Figure 3. Cell Reference Electrode Profiles

electrode is large and approximately 300 mV. This voltage degradation is in agreement with the results of other studies.

The half-cell reaction at the cadmium electrode is well known:



The $\text{Cd}(\text{OH})_2$ is also known to form through dissolution precipitation mechanism (Reference 7):



Reactions (1) and (2) precipitate cadmium hydroxide and migrate to the surface of the electrode towards the separator and the positive electrode. In the initial stages of cycling, the cadmium hydroxide is present as a thin film masking some areas of active cadmium. As the cycling continues, these films provide convenient sites for crystal growth and mask the active cadmium surface with large crystals of cadmium hydroxide. Scanning electron microscope (SEM) pictures of the negative electrode of cells exhibiting a voltage plateau revealed very large crystals of cadmium hydroxide; however, the positive plate did not show any crystal growth. Ford (Reference 8) demonstrated that the nickel-cadmium cells with electrolyte starvation suffered both in voltage and capacity. The cells under investigation were Gulton 6-Ah cells cycled at 20°C, 25 percent DOD with a voltage limit of 1.417 and a C to D ratio of approximately 1.15. By increasing the electrolyte content, the capacity degradation of these cells was overcome but the voltage plateau effect still existed. An increase of electrolyte appears to dissolve the smaller crystals but a bulk of large crystals masks the active surface of the electrode. Recent experiments show that continuous charging of negative

electrodes with large crystal growth in flooded condition almost eliminates the crystals. SEM pictures of negative electrodes, after gassing by overcharging in flooded cells, did not show any traces of large crystals. This finding agrees with the results of Fritzwill and Hess (Reference 9) who pointed out that prolonged reduction leads to the dissolution of crystals of $\text{Cd}(\text{OH})_2$.

Furthermore, the tear-down analysis of cycled cells has shown that cadmium migration is very heavy in areas under compression and that oftentimes the separator sticks strongly to the electrode surface. This occurrence may be explained by assuming that electrolytes are squeezed out from areas of increased compression, and these electrolyte starved areas seem to be good nucleation sites for crystal growth. The crystal size grows as the cycling continues and finally results in loss of voltage and capacity. A situation may occur when the cadmium dendrites will pass through the weak separator and cause soft or hard shorts that result in cell failure. If cadmium migration and large crystal growth can be suppressed, then the voltage degradation and plateau observed during cycling may be reduced.

CONCLUSIONS

The following points were concluded:

- The second voltage plateau is exhibited mainly by the negative electrode.
- The formation of large crystals of $\text{Cd}(\text{OH})_2$ on the negative electrode during cycling is responsible for voltage degradation.
- The continuous gassing of the negative electrode in flooded cells leads to the dissolution of large crystals of cadmium hydroxide.

Efforts to establish conditions that will minimize cadmium migration will be studied and may include the use of a thin-film coating, electroplating, or additives to achieve this purpose. In addition, plans are underway to study the effect of varying plate-stack compression on cadmium migration.

CADMIUM MIGRATION

INTRODUCTION

As demands for longer lifetime and more reliable space batteries increase, understanding and solving cell aging factors and mechanisms becomes essential. The primary effort has been aimed toward increasing cell performance, capacity, and lifetime, without concern about the basic electrode and side reactions. Some doubts still exist about the nature of the charge and discharge reactions in the positive and negative electrodes, the mechanism of oxygen recombination, the distribution of electrolyte, and the morphology of the active material with aging. Certain factors seem to limit the performance and the lifetime of the cell. These factors include negative fading, cadmium migration, positive-plate expansion and deterioration, loss of electrolyte and separator dryout, undesirable pressure buildup, and carbonate contamination. The object of this study is to examine one of the degradation mechanisms in the cell (i.e., the migration of cadmium hydroxide from the negative electrode into or toward the separator area.) It is difficult to determine any one factor as the cause of cadmium migration; however, circumstances or conditions occur in which migration is enhanced. These circumstances include the extent and the nature of the cycle regimes, temperature, the extent of carbonate contamination, and the nature and condition of the separator. The purpose of the present study effort is to investigate the causes of cadmium migration as a function of charge and discharge mode, rate of charge and discharge, and temperature.

EXPERIMENTAL METHOD

To study cadmium migration, a test matrix of charge and discharge rate, temperature, and time duration was developed. See Figure 4.

	10°C	25°C	40°C
C/3	24 hrs	48 hrs	72 hrs
C/5	72 hrs	24 hrs	48 hrs
C/10	48 hrs	72 hrs	24 hrs

Figure 4. Cadmium Migration Test Matrix

Initially, cadmium was measured in the separator, the positive electrode, and the electrolyte. Flooded cells were assembled, charged according to the matrix, and reanalyzed for cadmium in the cell components. Data from this analysis have been averaged and are summarized in Tables 6 through 9.

RESULTS AND DISCUSSION

The preliminary results on cadmium migration indicate that the positive electrode gains cadmium as a result of increasing the charge rate and charge time. Increasing the temperature enhances the rate of cadmium migration.

Table 6
Change in Concentration of Cadmium
in Positive Plate During Charge Mode

Charge Rate	C/3	C/5	C/10
$\Delta(\text{Cd})$ in mg	0.43	0.89	1.4
Temperature $\Delta(\text{Cd})$ in mg	10°C 0.45	25°C 0.88	40°C 1.4
Duration $\Delta(\text{Cd})$ in mg $\Delta(\text{Cd})/\text{day}$	24 hrs 0.39 0.39	48 hrs 0.83 0.42	72 hrs 1.5 0.50

Table 7
Change in Concentration of Cadmium
in Electrolyte During Charge Mode

Charge Rate	C/3	C/5	C/10
$\Delta(\text{Cd})$ in mg near positive $\Delta(\text{Cd})$ in mg near negative	-- -0.20	0.02 -0.02	0.01 -0.13
Temperature $\Delta(\text{Cd})$ in mg near positive $\Delta(\text{Cd})$ in mg near negative	10°C -0.02 -0.09	25°C -0.04 -0.10	40°C -- -0.17
Duration $\Delta(\text{Cd})$ in mg near positive $\Delta(\text{Cd})$ in mg near negative	24 hrs -0.02 -0.10	48 hrs -- -0.15	72 hrs 0.01 -0.10

Table 8
Change in Concentration of Cadmium
in Positive Plate During
Discharge Mode

Discharge Rate	C/3	C/5	C/10
$\Delta(\text{Cd})$ in mg	11.7	9.0	5.5
Temperature	10°C	25°C	40°C
$\Delta(\text{Cd})$ in mg	3.8	9.8	12.5
Duration of discharge	24 hrs	48 hrs	72 hrs
$\Delta(\text{Cd})$ in mg	5.4	10.2	10.5

Table 9
Change in Concentration of Cadmium
in Electrolyte During Discharge

Charge Rate	C/3	C/5	C/10
$\Delta(\text{Cd})$ in mg near positive	0.0	0.05	0.06
$\Delta(\text{Cd})$ in mg near negative	-0.29	-0.38	-0.58
Temperature	10°C	25°C	40°C
$\Delta(\text{Cd})$ in mg near positive	0.07	0.08	0.04
$\Delta(\text{Cd})$ in mg near negative	-0.39	-0.65	-0.31
Duration	24 hrs	48 hrs	72 hrs
$\Delta(\text{Cd})$ in mg near positive	0.16	0.01	-0.06
$\Delta(\text{Cd})$ in mg near negative	-0.02	-0.48	-0.84

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